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Indian Standard
SPECIFICATION FOR *p*-NITROPHENOL

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Indian Standard

SPECIFICATION FOR *p*-NITROPHENOL

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Indian Standard

SPECIFICATION FOR *p*-NITROPHENOL

0. FOREWORD

0.1 This Indian Standard was adopted by the Indian Standards Institution on 29 June 1979, after the draft finalized by the Organic Chemicals (Miscellaneous) Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 *p*-Nitrophenol is used in the manufacture of rhodamine dyestuffs, phenolphthalein, methyl parathion, etc. It is also used as an indicator in alkalimetry and for the testing of blood and seawater.

0.3 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

1. SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for *p*-nitrophenol.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of light yellow to yellow brown moist crystals.

2.2 The material shall also comply with the requirements given in Table 1 when tested according to the methods prescribed in Appendix A. References to the relevant clauses of Appendix A are given in col 4 of the table.

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in mild steel drums with polyethylene or other suitable lining.

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*Rules for rounding off numerical values (*revised*).

TABLE 1 REQUIREMENTS FOR *p*-NITROPHENOL (ON DRY BASIS)

(*Clause* 2.2)

Sl No.	Characteristic	Requirement	Method of Test (Ref to Cl No. in Appendix A)
(1)	(2)	(3)	(4)
i)	*p*-Nitrophenol content, percent by mass, *Min*	99	A-2
ii)	Melting point, °C, *Min*	112	A-3
iii)	Moisture, percent by mass, *Max*	8	A-4
iv)	Sulphated ash, percent by mass, *Max*	0·1	A-5
v)	Insoluble matter, percent by mass, *Max*	0·5	A-6
vi)	*p*-Nitrochlorobenzene (PNCB) content	Nil	A-7
vii)	*o*-Nitrophenol content, percent by mass, *Max*	0·2	A-7

3.2 Marking — The containers shall be securely closed and marked legibly and indelibly with the following information:

a) Name of the material;

b) Mass of the material in the container;

c) Name of the manufacturer and his recognized trade-nark, if any; and

d) Lot or batch number in code or otherwise.

3.2.1 The containers may also be marked with the ISI Certification Mark.

NOTE — The use of the ISI Certification Mark is governed by the provisions of the Indian Standards Institution (Certification Marks) Act and the Rules and Regulations made thereunder. The ISI Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well-defined system of inspection, testing and quality control which is devised and supervised by ISI and operated by the producer. ISI marked products are also continuously checked by ISI for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the ISI Certification Mark may be granted to manufacturers or processors, may be obtained from the Indian Standards Institution.

4. SAMPLING

4.1 The method of drawing representative test samples of the material and the criteria for conformity shall be as prescribed in Appendix B.

APPENDIX A

(*Clause* 2.2, and *Table* 1)

ANALYSIS OF *p*-NITROPHENOL

A-1. QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be used in tests.

NOTE — ' Pure chemicals ' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2. DETERMINATION OF *p*-NITROPHENOL CONTENT

A-2.0 Methods — Two methods, namely, Method A and Method B, have been prescribed. In case of dispute, Method B shall be used as a referee method.

A-2.1 Method A (Bromination Method)

A-2.1.0 *General* — The method is based on the bromination of sodium salt solution of *p*-nitrophenol. The brominating reagent employed is standard bromate-bromide solution.

A-2.1.1 *Reagents*

A-2.1.1.1 *Standard potassium bromate-bromide solution* — 0·1 N. Dissolve 2·784 g of potassium bromate and 12 g of potassium bromide in 1 000 ml of water.

A-2.1.1.2 *Standard sodium thiosulphate solution* — 0·1 N.

A-2.1.1.3 *Sodium hydroxide solution* — 1 N.

A-2.1.1.4 *Potassium iodide solution* — 10 percent (*m/v*).

A-2.1.1.5 *Concentrated hydrochloric acid* — conforming to IS : 265-1976†.

A-2.1.1.6 *Starch solution*

A-2.1.2 *Procedure* — Transfer 6 to 9 g of accurately weighed test substance with cold water into a 500-ml volumetric flask and dissolve by adding 55 ml of sodium hydroxide solution and hot water. After cooling to room temperature, make up to the mark with water. To 10 ml of this solution add 50 ml of potassium bromate-bromide solution and 25 ml of hydrochloric acid. Stopper the flask and allow it to stand for 15 minutes at room temperature. Back-titrate the excess of potassium bromate-bromide solution with standard sodium thiosulphate solution after adding 25 ml of potassium iodide solution, using starch as indicator.

---

*Specification for water for general laboratory use (*second revision*).

†Specification for hydrochloric acid (*second revision*).

Pipette 50 ml of potassium bromate-bromide solution in iodometric flask and add 25 ml of concentrated hydrochloric acid and 25 ml of potassium iodide solution, wash the sides of the flask with little water and titrate the liberated iodine against standard sodium thiosulphate, using starch as indicator.

A-2.1.3 *Calculation*

$$p\text{-Nitrophenol content, percent by mass} = \frac{(V_1 - V_2) \times N \times \text{mol mass} \times 1{\cdot}25}{M}$$

where

V_1 = volume in ml of standard sodium thiosulphate solution used in the blank titration,

V_2 = volume in ml of standard sodium thiosulphate used in the titration,

N = normality of standard sodium thiosulphate solution,

mol mass = gram molecular mass of the material, and

M = mass in g of the material taken for the test.

A-2.1.3.1 The value calculated as above on *as received basis* shall be corrected on *dry basis.*

A-2.2 Method B (Titanium Sulphate Method) — The principle involved in this method is the estimation of total nitro function by reduction using titanium (III) sulphate.

A-2.2.1 *Reagents*

A-2.2.1.1 *Glacial acetic acid*

A-2.2.1.2 *Sodium acetate solution* — 2 N.

A-2.2.1.3 *Titanium (III) sulphate solution* — Approximately 0·5 N. Boil 360 ml of 15 percent titanous sulphate solution with 3 to 4 N sulphuric acid. Cool it under nitrogen atmosphere and then make up to 500 ml with sulphuric acid. This solution shall be stored in amber-coloured bottle.

A-2.2.1.4 *Hydrochloric acid* — *See* IS : 265-1976*.

A-2.2.1.5 *Standard ferric ammonium sulphate solution* — 0·2 N. Dissolve 76·42 g of pure ferrous ammonium sulphate [$FeSO_4.(NH_4)_2SO_4.6H_2O$] in 500 ml of water. Add 80 ml of concentrated sulphuric acid and mix well. Dissolve 6·32 g of potassium permanganate in 100 ml of water.

---

*Specification for hydrochloric acid (*second revision*).

Add this solution to the solution of ferrous ammonium sulphate with stirring. At the end, add the permanganate solution carefully so that ferrous salt is just oxidized and a faint pink colour develops. Cool the solution and dilute it to 1 litre. Determine the normality of the solution by the method given in Appendix B of IS : 3859-1966*.

A-2.2.1.6 *Aqueous ammonium thiocyanate solution* — 10 percent.

A-2.2.2 *Procedure* — Dissolve 6·9 g of the test substance in glacial acetic acid and make up to 500 ml. Add 25 ml of the above solution to a 500-ml flat-bottomed flask, followed by 25 ml of glacial acetic acid and 50 ml of sodium acetate solution. Start flushing the flask and pipette simultaneously with oxygen-free nitrogen or carbon dioxide as described in IS : 3859-1966*. Add 40 ml of titanium sulphate solution to the flask. Allow 2 to 3 minutes to complete the reduction. Back-titrate the excess of titanium sulphate solution with ferric ammonium sulphate solution after acidifying the mixture with 25 ml of concentrated hydrochloric acid, using 10 ml of ammonium thiocyanate as indicator. Also carry out a blank determination using 20 ml of titanium sulphate solution and excluding the sample solution.

A-2.2.3 *Calculation*

$$p\text{-Nitrophenol content, percent by mass} = \frac{(2V_1 - V_2) \times N \times D \times 0{\cdot}023\,17 \times 100}{6{\cdot}9}$$

where

V_1 = volume in ml of standard ferric ammonium sulphate solution used in the blank titration,

V_2 = volume in ml of standard ferric ammonium sulphate solution used in the back titration,

N = normality of standard ferric ammonium sulphate solution, and

D = dilution factor = 20.

A-3. DETERMINATION OF MELTING POINT

A-3.1 Procedure — Carry out the determination of melting point according to the method prescribed in IS : 5762-1970†.

A-4. DETERMINATION OF MOISTURE CONTENT

A-4.0 Outline of the Method — Azeotropic dehydration using toluene as distillation agent and measurement of volume of water after condensation and decantation.

---

*Method for determination of strength of water soluble azo dyes by reduction with titanium trichloride.

†Method for the determination of melting point and melting range.

A-4.1 Reagents

A-4.1.1 *Toluene* — *See* IS : 1839-1961*.

A-4.2 Apparatus — The apparatus is essentially a ground-glass flask of 500 ml capacity, fitted with a reflux condenser and a device permitting to condense the vapours and measure the volume of condensed water in a graduated collector. A standard Dean and Stark Assembly (*see* Fig. 1) with a 10-ml receiver (*see* Fig. 2) is suitable for the purpose.

A-4.3 Procedure — Weigh accurately about 25 g of the material in the flask. Add 120 ml of toluene. Assemble the apparatus as shown in Fig. 1. Heat gently at the boiling point, so that the rate at which the condensate falls from the end of the condenser is 2 to 5 drops per second. Continue the distillation until condensed water is no longer visible in any part of the apparatus except the bottom of the graduated tube, and until the volume of water collected remains constant. Leave at rest for 5 minutes. Read the volume of water collected in the receiver.

A-4.4 Calculation

$$\text{Moisture, ml/100 g} = \frac{V \times 100}{M}$$

where

V = volume in ml of water collected in receiver, and

M = mass in g of the material taken for the test.

A-5. DETERMINATION OF SULPHATED ASH

A-5.1 Apparatus

A-5.1.1 *Platinum Crucible*

A-5.1.2 *Muffle Furnace* — capable of maintaining a temperature of 900 ± 25°C.

A-5.2 Reagents

A-5.2.1 *Concentrated Sulphuric Acid* — conforming to IS : 266-1977†.

A-5.2.2 *Concentrated Nitric Acid* — conforming to IS : 264-1976‡.

A-5.3 Procedure — Weigh accurately about 1 to 1·2 g of the material in a silica or platinum crucible. Heat the crucible on a low bunsen burner flame so that moisture of the *p*-nitrophenol sublimes away. Cool to room temperature and add 1 ml of 1 : 1 mixture of sulphuric acid and nitric acid mixture. Heat once again on a low bunsen burner flame. After all the acid has evaporated heat the crucible on a strong flame. Transfer the crucible to preheated muffle furnace and keep there for 15 minutes. Cool to room temperature and weigh.

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*Specification for toluene, reagent grade.

†Specification for sulphuric acid (*second revision*).

‡Specification for nitric acid (*second revision*).

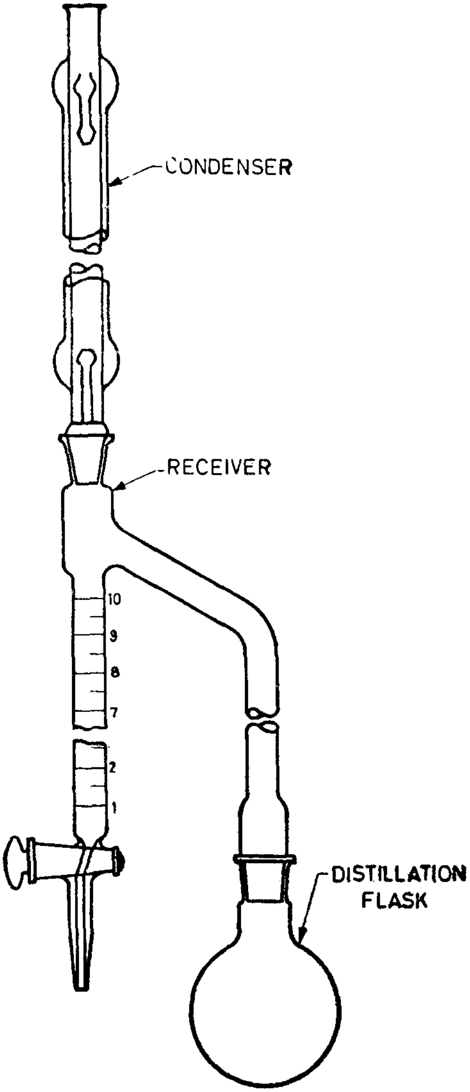
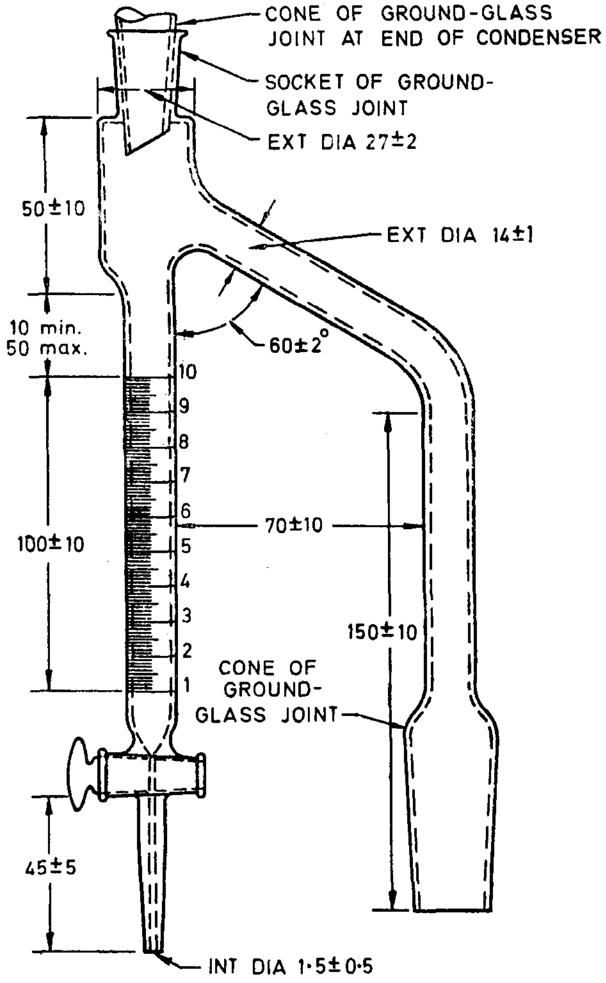


FIG. 1 DEAN AND STARK ASSEMBLY


CONE OF GROUND-GLASS JOINT AT END OF CONDENSER
SOCKET OF GROUND-GLASS JOINT
EXT DIA 27±2
50±10
EXT DIA 14±1
10 min.
50 max.
60±2°
10
9
8
7
6
5
4
3
2
1
100±10
70±10
150±10
CONE OF GROUND-GLASS JOINT
45±5
INT DIA 1·5±0·5

All dimensions in millimetres.

FIG. 2 10-ml RECEIVER (DEAN AND STARK APPARATUS)

A-5.4 Calculation

$$\text{Sulphated ash, percent by mass} = \frac{100\,M_1}{M_2}$$

where

M_1 = mass in g of the ignited residue, and
M_2 = mass in g of the material taken for the test.

A-6. DETERMINATION OF MATTER INSOLUBLE IN WATER

A-6.1 Reagent

A-6.1.1 *Sodium Hydroxide Solution* — 5 percent.

A-6.2 Procedure — Weigh 10·000 g of the material in a beaker and heat with sodium hydroxide solution to form sodium salt of *p*-nitrophenol. Add sufficient quantity of water till it forms a clear yellow solution. Filter through a previously dried and weighed sintered-glass crucible G 4 and wash the residue with water. Dry the crucible with the residue to constant mass at 105 to 110°C.

A-6.3 Calculation

$$\text{Matter insoluble in water, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of the crucible along with the residue,
M_2 = mass in g of the crucible, and
M = mass in g of the sample taken for the test.

A-7. TEST FOR *p*-NITROCHLOROBENZENE AND *o*-NITROPHENOL CONTENT

A-7.1 Reagents

A-7.1.1 *Acetone* — dry and redistilled chromatographic grade.

A-7.1.2 *Silica Gel G* — Passing completely through 150-micron IS Sieve.

A-7.1.3 *Eluent* — Ligroin and acetone in the ratio of 97·5 : 2·5 (*v/v*).

A-7.1.4 *Titanium Sulphate Solution* — 0·05 N.

A-7.2 Test Substances — The following components are estimated:

a) *p*-nitrochlorobenzene
b) *m*-nitrochlorobenzene
c) *o*-nitrophenol

A-7.3 Procedure

A-7.3.1 *Test Solution* — Dissolve 10·00 g of the test substance in acetone, make up to 100 ml.

A-7.3.2 *Reference Solutions*

A-7.3.2.1 Dissolve 0·500 g each of pure *o*-nitrophenol, *m*-, and *p*-nitrochlorobenzenes in acetone and make up to 250 ml. Mark it as *Solution A.*

A-7.3.2.2 Take 50 ml of Solution A and dilute with acetone making up the level to 100 ml. Mark it as *Solution B.*

A-7.3.2.3 Weigh 10·00 g of pure *p*-nitrophenol into six 100-ml measuring flasks with grould stoppers. Then add to each flask the under mentioned quantities of solution A or B, using pipettes. If necessary, dissolve the *p*-nitrophenol by addition of further acetone, and make up the level with acetone. Keep the reference solutions ready in amber-coloured reagent bottles with ground stoppers.

75 ml solution A = 1·5 percent each of *o*-nitrophenol and *m*-, *p*-nitrochlorobenzene
50 ml solution A = 1·0 ,,
25 ml solution A = 0·5 ,,
25 ml solution B = 0·25 ,,
10 ml solution B = 0·10 ,,
5 ml solution B = 0·05 ,,

A-7.3.3 *Quantities to be Used* — 5 ml each.

A-7.3.4 *Conditions*

Duration	45 to 60 minutes, approx
Elution	17 cm, approx
Temperature	20± 2°C

A-7.3.5 *Evaluation* — The following steps are taken for evaluating the impurities:

a) Reduction of the nitro-compounds by spraying with titanium sulphate reagent and drying at room temperature for 20 to 30 minutes, under the hood.

b) Diazotization with nitrous fumes. Spray the plate slightly with concentrated hydrochloric acid and then keep it in an atmosphere of nitrous fumes. After approximately 5 minutes, take the plate out and blow with a warm air-blower to remove nitrous fumes and hydrochloric acid in excess. The layer shall dry only on the surface, not entirely.

c) Coupling by spraying with N-(1-naphthyl)-ethylenediammoniumdichloride solution. The spot corresponding to *o*-nitrophenol develops the maximum colour intensity only after approximately 30 to 60 minutes. The other substances, however, can be estimated shortly after coupling.

A-7.4 Evaluation Order

a) *p*-nitrochlorobenzene
b) *m*-nitrochlorobenzene
c) *o*-nitrophenol

A-7.5 Calculation

$$\frac{a \times 40 \times 69 \times 1\,000}{30\,000 \times 13{\cdot}8} = a \times 0{\cdot}666\,7 = A \text{ (from } NO_2 \text{)}$$

$$\frac{(\,d + e\,) \times 69}{157{\cdot}5} = (\,d + e\,) \times 0{\cdot}438 = C = \text{g nitrite for } m\text{- and } p\text{-nitrochlorobenzene}$$

$$A - C = D = \text{g nitrite for } o\text{- and } p\text{-nitrophenol/100 g (from } NO_2 \text{)}$$

$$D \times \frac{139}{69} = D \times 2{\cdot}014 = \text{percent nitrophenol (from } NO_2 \text{)}$$

where

a = volume in ml of standard titanium sulphate solution used in the reduction,

d = percent of *m*-nitrochlorobenzene (reading taken from the chromatagram), and

e = percent of *p*-nitrochlorobenzene (reading from chromatagram).

APPENDIX B

(*Clause* 4.1)

SAMPLING OF *p*-NITROPHENOL

B-1. SCALE OF SAMPLING

B-1.1 Lot — In a single consignment, all the drums of the material belonging to the same batch of manufacture shall constitute a lot.

B-1.2 For ascertaining the conformity of the material in the lot, to the requirements of the specification, samples shall be tested for each lot separately.

B-1.3 The number of drums to be selected from the lot shall depend on the size of the lot and shall be in accordance with Table 2.

TABLE 2 SCALE OF SAMPLING

No. of Drums in the Lot N	Sample Size n
(1)	(2)
Up to 25	3
26 ,, 50	4
51 ,, 100	5
101 ,, 300	6
301 ,, 500	7
501 and above	8

B-1.3.1 The drums shall be selected at random. In order to ensure the randomness of selection, a random number table shall be used. For guidance and use of random number tables, IS : 4905-1968* may be referred. In the absence of a random number table, the following procedure may be adopted:

> Starting from any drum in the lot, count them at 1, 2, 3,.........up to r and so on in one order, where r is an integral part of N/n. Every rth drum thus counted shall be withdrawn to constitute the sample.

B-2. TEST SAMPLES AND REFEREE SAMPLE

B-2.1 Draw with an appropriate sampling instrument a small portion of the material from different parts of each drum selected according to **B-1.3.** The total quantity of material drawn from each drum shall be sufficient to make triplicate determinations for all the characteristics given in the specification.

B-2.2 Thoroughly mix all portions of the material drawn from the same drum. Out of these portions, equal quantity of material shall be taken from each selected drum and shall be well mixed up together so as to form a composite sample. The quantity of material for the composite sample shall be sufficient to make triplicate determinations for all the characteristics tested on the composite sample (*see* **B-3.2.**). The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

---

*Methods for random sampling.

B-2.3 The remaining portion of the material from each container (after the quantity needed for the composite sample has been taken), shall be divided into three equal parts. These parts shall be immediately transferred to separate bottles and labelled with all the particulars of sampling. The material in each such bottle shall constitute an individual test sample. These individual samples shall be separated into three identical sets of samples in such a way that each set has an individual test sample representing each container selected. One of these three sets shall be sent to the purchaser, another to the supplier and the third shall be used as referee sample.

B-2.4 Referee Sample — The referee sample shall consist of the composite samples (*see* **B-2.2**) and a set of individual samples (*see* **B-2.3**) marked for this purpose and shall bear the seals of the purchaser and the supplier. These shall be kept at a place agreed to between the purchaser and the supplier and shall be used in case of a dispute between the two.

B-3. NUMBER OF TESTS

B-3.1 *p*-Nitrophenol content shall be tested on each of the individual samples.

B-3.2 Tests for all other requirements given in **2** of the specification shall be carried out on the composite sample.

B-4. CRITERIA FOR CONFORMITY

B-4.1 Individual Samples — For *p*-nitrophenol content, the mean and range of the test results shall be computed as follows:

$$\text{Mean } (X) = \frac{\text{sum of individual test results}}{\text{Number of tests}}$$

$$\text{Range} (R) = \text{difference between the maximum and minimum values of test results.}$$

B-4.2 The lot shall be declared to have satisfied the requirement for *p*-nitrophenol content if the expression $X - 0{\cdot}6\ R$ is greater than or equal to 98.

B-4.3 Composite Sample — For declaring the conformity of the lot to the requirements of all other requirements of the specification, all the test results on the composite sample shall meet the corresponding specification requirements.

B-4.4 The lot shall be declared as conforming to the requirements of the specification, if **B-4.2** and **B-4.3** are satisfied.

(*Continued from page* 2)

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